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☐ 1.   4/34/1 (Item 1 from file: 351)

012523932  
 WPI Acc No: 1999-330038/ 199928  
 Polyester fibre web for garments, filter, tape etc -  
 consists of extra fine fibre made of polytrimethylene terephthalate  
 having predefined mean diameter and intrinsic viscosity  
 Patent Assignee: ASAHI KASEI KOGYO KK (ASAHI)  
 Number of Countries: 001   Number of Patents: 001  
 Patent Family:  

Patent No	Kind	Date	Applicat No	Kind	Date	Week
JP 11107154	A	19990420	JP 97279377	A	19970929	199928   B

 Priority Applications (No Type Date): JP 97279377 A 19970929  
 Patent Details:  

Patent No	Kind	Lan	Pg	Main IPC	Filing Notes
JP 11107154	A		6	D04H-003/16	

 Abstract (Basic): JP 11107154 A  
 NOVELTY - The web consists of extra fine fibers made of  
 polytrimethylene terephthalate having mean diameter of 0.8-5  $\mu$ m and  
 intrinsic viscosity of 0.4-1.0.  
 USE - For garments, artificial leather base fabric, filter, tape,  
 disposable diaper, wiping cloth etc.  
 ADVANTAGE - The fiber web has excellent softness and wear property.  
 Dwg.0/1  
 Derwent Class: A84; A88; A96; D22; F04; J01  
 International Patent Class (Main): D04H-003/16  
 International Patent Class (Additional): D01F-006/62

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☐ 2.   4/34/2 (Item 2 from file: 347)  
 06165608 POLYESTER ULTRAFINE FIBER WEB

Pub. No.: 11-107154 A ]  
 Published: April 20, 1999 (19990420)  
 Inventor: KATO JINICHIRO  
           FUJIMOTO KATSUHIRO  
 Applicant: ASAHI CHEM IND CO LTD

Application No.: 09-279377 [JP 97279377]  
Filed: September 29, 1997 (19970929)  
International Class: D04H-003/16; D01F-006/62

## ABSTRACT

**PROBLEM TO BE SOLVED:** To obtain a polytrimethylene terephthalate ultrafine fiber web by melt blow method, excellent in softness and abrasion property, capable of dyeing into deep color at normal pressure and hardly containing polymer balls.

**SOLUTION:** This polyester ultrafine fiber web comprises an ultrafine fiber web composed of substantially polytrimethylene terephthalate comprising an ultrafine fiber group having 0.8–5  $\mu\text{m}$  average fiber diameter and 0.4–1.0 intrinsic viscosity. The resultant polytrimethylene terephthalate ultrafine fiber web is useful for clothes for base fabric or the like of synthetic leather substitutes, filters, tapes, disposable diapers, base fabric of catapasm, fatigue uniforms, wiping clothes, heat-insulating cotton pad, core fabrics, sheet or the like.

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☐ 3.

4/34/3 (Item 3 from file: 345)

15275575

Basic Patent (No,Kind,Date): JP 11107154 A2 990420

### PATENT FAMILY:

JAPAN (JP)

Patent (No,Kind,Date): JP 11107154 A2 990420

POLYESTER ULTRAFINE FIBER WEB (English)

Patent Assignee: ASAHI CHEMICAL IND

Author (Inventor): KATO JINICHIRO; FUJIMOTO KATSUHIRO

Priority (No,Kind,Date): JP 97279377 A 970929

Applic (No,Kind,Date): JP 97279377 A 970929

IPC: \* D04H-003/16; D01F-006/62

CA Abstract No: \* 130(23)313163P; 130(23)313163P

Language of Document: Japanese

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☐ 1. 2/34/1 (Item 1 from file: 351)

008274474

WPI Acc No: 1990-161475/ 199021

Fine polyester fibre web - comprises polyethylene terephthalate and is obtd. by melt-blowing polyester

Patent Assignee: ASAHI CHEM IND CO LTD (ASAH )

Number of Countries: 001 Number of Patents: 001

Patent Family:

Patent No	Kind	Date	Applicat No	Kind	Date	Week
JP 2104755	A	19900417				199021 B

Priority Applications (No Type Date): JP 7946037 A 19790417; JP 89192716 A 19890101

Abstract (Basic): JP 2104755 A

A web consisting of a very fine polyester fibre is obtd. by melt-blowing of a polyester polymer and the very fine fibre has ave. fibre 0.8-5.0 mm and has intrinsic viscosity 0.45-0.80. The very fine web is composed of polyethylene terephthalate. A web consisting of a very fine polyester fibre in which Y value of the fibre web is at 2.5-7.0. The polyester polymer to compose the polyester fibre has intrinsic viscosity 0.50-0.90 and the intrinsic viscosity of the fine fibre (A) and a polyester polymer (B) satisfy the equation: A is greater than or equal to B - 0.2. When the veyr fine fibre is measured by differential scanning colorimetry(DSC), crystallisation peak is small and melting peak is multiple. Unevenness of web wt. in the width direction is less than 10%. The web is not polymer nap.

USE/ADVANTAGE - The web has good strength and dyeing properties and is pref. used as a ground cloth for a raised man-made leather. (8pp Dwg.No.0/2)

Derwent Class: A23; F04

International Patent Class (Additional): D04H-001/72

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☐ 2. 2/34/2 (Item 2 from file: 347)

03129255 \*\*Image available\*\*

ULTRAFINE FIBER WEB OF POLYESTER

Pub. No.: 02-104755 [JP 2104755 A]

Published: April 17, 1990 (19900417)

Inventor: IKEDA MASATAKA

ISHIKAWA TAKEO

SHIMA TSUKASA

Applicant: ASAHI CHEM IND CO LTD [000003] (A Japanese Company or Corporation), JP (Japan)

Application No.: 01-192716 [JP 89192716]

Filed: July 27, 1989 (19890727)

International Class: [ 5 ] D04H-001/72; D04H-001/42

JAPIO Class: 15.2 (FIBERS -- Cloth Products)

JAPIO Keyword: R057 (FIBERS -- Non-woven Fabrics)

Journal: Section: C, Section No. 736, Vol. 14, No. 310, Pg. 86, July 04, 1990 (19900704)

#### ABSTRACT

PURPOSE: To obtain the title web suitable as ground fabric napped artificial leather having specific average fiber diameter and specific intrinsic viscosity, tenacity, excellent dyeability, flexibility and a little occurrence of polymer pilling, comprising a polyester polymer by melt blowing method.

CONSTITUTION: A polyester polymer (preferably polyethylene terephthalate) is melted by an extruder 1, extruded from a spinning orifice of a die 2 while blowing a high-temperature air flow upon the as-spun fibers to give the aimed web 5 having 0.8-5.0.mu.m average fiber diameter, 0.45-0.80, preferably 0.50-0.70 intrinsic viscosity and preferably 3.0-6.0Y value. When intrinsic viscosity of ultrafine fiber  $\geq$  intrinsic viscosity of polyester-0.2 is satisfied, web having excellent weight distribution in the width direction is obtained.

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☐ 3. 2/34/3 (Item 3 from file: 345)

9258193

Basic Patent (No,Kind,Date): JP 2104755 A2 900417

#### PATENT FAMILY:

JAPAN (JP)

Patent (No,Kind,Date): JP 2104755 A2 900417

ULTRAFINE FIBER WEB OF POLYESTER (English)




Patent Assignee: ASAHI CHEMICAL IND


Author (Inventor): IKEDA MASATAKA; ISHIKAWA TAKEO; SHIMA TSUKASA  
Priority (No,Kind,Date): JP 89192716 A 890727  
Applic (No,Kind,Date): JP 89192716 A 890727  
IPC: \* D04H-001/72; D04H-001/42  
Derwent WPI Acc No: ; C 90-161475  
JAPIO Reference No: ; 140310C000086  
Language of Document: Japanese

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CLAIMS

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[Claim(s)]

[Claim 1] The polyester super-thin fiber web which is a super-thin fiber web which consists of polytrimethylene terphthalate substantially, and serves as a 0.8-5-micrometer diameter of average fiber from the super-thin fiber group which has the limiting viscosity of 0.4-1.0.

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[Translation done.]

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention relates to the polytrimethylene terephthalate super-thin fiber web by the melt blowing method. Furthermore, in detail, it excels in flexibility and wear nature, and can be dyed a dark color by the ordinary pressure, and a polymer ball is related with the polytrimethylene terephthalate super-thin fiber web by the melt blowing method.

[0002]

[Description of the Prior Art] About the spinning method of the polymer by the melt blowing method Industrial - and - engineering chemistry The 48th volume, No. 8, 1956 It is indicated by 1342-1346 pages and the super-thin fiber web which used the polyethylene terephthalate in it is introduced. It or subsequent ones, a polyethylene-terephthalate homopolymer, a polyethylene-terephthalate copolymer, Many patent application about the super-thin fiber web by the melt blowing method using the polybutylene terephthalate is made (JP,53-65471,A, JP,63-53309,B, JP,3-8855,A, JP,4-2850,A).

[0003] The polytrimethylene terephthalate which is made to carry out the polycondensation of the trimethylene glycol (1, 3-propanediol) to the lower-alcohol ester of the terephthalic acid represented by a terephthalic acid or the dimethyl terephthalate on the other hand, and is obtained is the epoch-making polymer having the outstanding elastic-recovery nature, a low elastic modulus (soft feeling), a property similar to a polyamide called dyeable, and lightfastness, heat set nature, dimensional stability and a performance similar to a polyethylene terephthalate called a low water absorption. Taking advantage of the feature, it is applied to the BCF carpet, the brush, the tennis gut, etc. (JP,9-3724,A, JP,8-173244,A, JP,5-262862,A).



[0004] If the melt blow super-thin fiber web using polytrimethylene terephthalate is obtained, although a web with the new feature is expectable, the present condition is that such [ until now ] a proposal is not made. Of course, in application of the polyester web by melt blow, the feature, and the industrial usefulness or industrial manufacturing method of a certain thing are not shown for what is shown as one of the examples of polymer by which many TORITORI methylene terephthalate was enumerated as an example of polyester.

[0005]

[Problem(s) to be Solved by the Invention] It may be able to be imagined that it can carry out a melt blow easily on the conditions near a polyethylene terephthalate fundamentally since the molecular structure of polytrimethylene terephthalate is the structure which introduced one methylene into the ethylene portion of a polyethylene terephthalate. However, polytrimethylene terephthalate has difference, like thermal stability with a very quick crystal speed contains many oligomer and low-molecular-weight impurities in a low and a polymerization object considerably as compared with a polyethylene terephthalate, and only a crude web is obtained even if it applies the manufacture conditions in the case of being a polyethylene terephthalate as it is.

[0006] The technical problem of this invention is applying the outstanding performance which polytrimethylene terephthalate's has to a super-thin fiber web, pulling out a new performance, and establishing the industrial manufacturing method. Consequently, the polytrimethylene terephthalate super-thin fiber web was excellent in flexibility and wear nature, could be dyed the dark color by the ordinary pressure, and found out the bird clapper to the web with few polymer balls. Although the polyester super-thin fiber web by the melt blowing method for could dye it the dark color by the ordinary pressure, and moreover having excelled in robustness is very useful to simplification of a dyeing process etc., the present condition is that there is no such [ until now ] thing parenchyma.

[0007]

[Means for Solving the Problem] That is, this invention is a super-thin fiber web which consists of polytrimethylene terephthalate substantially, and offers the polyester super-thin fiber web which serves as a 0.8-5-micrometer diameter of average fiber from the super-thin fiber group which has the limiting viscosity of 0.4-1.0. The polymer used for this invention is polytrimethylene terephthalate which carries out the polycondensation of a terephthalic acid and the 1 and 3-propanediol substantially, and is obtained. Even if it is it a polytrimethylene terephthalate homopolymer that it is substantial in this invention, it is shown that you may be the polytrimethylene

terphthalate copolymer shown below. namely, the range which does not spoil the purpose and effect of this invention -- glycol components, such as acid components, such as an isophthalic acid, a succinic acid, an adipic acid, 2, 6-naphthalene dicarboxylic acid, and 5-sulfoisophtharate tetrabutyl POSUONIUM salt, and 1, 4-butanediol, 1, 6-hexandiol, cyclohexane dimethanol, epsilon-caprolactone, 4-hydroxybenzoic acid, polyoxy ethylene glycol, a polytetramethylene glycol, etc. -- less than [ 10wt% ] -- copolymerization may be carried out

[0008] Moreover, to polytrimethylene terphthalate, the need may be accepted, and various kinds of additives, for example, a flatting, a thermostabilizer, a defoaming agent, a ready coloring material, a flame retarder, an antioxidant, an ultraviolet ray absorbent, an infrared-absorption agent, a crystalline-nucleus agent, a fluorescent brightener, etc. may be copolymerized or mixed. As for the polymer used for this invention, it is desirable that the content of the oligomer of trimethyleneterephthalate is less than [ 3wt% ], it becomes possible to avoid an on-the-strength fall by this, and also required spinning stability is industrially securable. in addition, the oligomer to which 2-4 trimethyleneterephthalate units were usually connected with trimethyleneterephthalate oligomer -- it is -- a line -- you may be a cyclic structure even if it is structure

[0009] In exceeding 3wt(s)%, when carrying out spinning, oligomer deposits in the circumference of a nozzle, and it becomes easy to generate a polymer ball, for example. In order to perform spinning to stability for a long time, less than [ 1.5wt% ] is less than [ 1wt% ] desirable still more preferably. furthermore -- the point that the fluff of the obtained fiber decreases -- less than [ 0.5wt% ] -- less than [ 0.3wt% ] is desirable and still more preferably natural -- an ideal target is non-\*\*. In addition, a polymer ball is the cob-like polymer generated in the edge and center section of the ball-like polymer which has a diameter with a diameter [ of web composition fiber ] of about about 10 to 500 times, or fiber. After observing this polymer ball using a microscope, or giving the means of a press, a calender, and confounding processing and others to remaining as it is or a web for a web and raising the fiber density, it is detectable by dyeing this. If a polymer ball exists mostly, the use of the super-thin fiber web obtained will be restricted greatly, and it will become impossible especially as a base fabric for artificial leather using it.

[0010] Furthermore, into the polymer used for this invention, it is desirable that the content of the with a molecular weight of 300 or less organic substance is less than [ 1wt% ], and it does not color within the limits of this, or the performance of excelling in lightfastness can be secured. The with a molecular weight of 300 or less said here

organic substance is the organic substance by which copolymerization is not carried out to polymer. According to examination of this invention persons, as the with a molecular weight of 300 or less organic substance, allyl alcohol, an acrolein, 2-butanol, a hexanol, a heptanol, a glycidyl methyl ether, the oxy-propylmethyl ether, etc. existed, and it found out that the total amount of these compounds had big influence on a moldability, product durability, and weatherability. When the content of the with a molecular weight of 300 or less organic substance exceeds 1wt%, it will become or a polymer ball will become being easy to generate what it is easy to color with light. Preferably, the content of the with a molecular weight of 300 or less organic substance is 5000 ppm or less, and is 1000 ppm or less especially preferably. Of course, it is non-<sup>\*\*</sup> ideally.

[0011] As the melting point of the polymer used for this invention, it is desirable that it is 227 degrees C or more. With the melting point, it is defined in 220-250 degrees C as the peak value of the peak considered to be dissolution here. When two or more dissolution peaks exist (a shoulder peak is also included), let the peak of low temperature be the melting point. At less than 227 degrees C, a weatherproof fall becomes easy for the melting point to take place. For example, if polytrimethylene terphthalate is manufactured at once and solid state polymerization of the polymer is carried out at about 200 degrees C, the content of oligomer can be reduced greatly. However, if solid state polymerization is performed, the melting point of raw material polymer falls greatly, and will be in the state where no less than 225 degrees C are not fulfilled. Into such polymer, copolymerization of the screw-3-hydroxypropyl ether which a trimethylene glycol dimerizes and generates is carried out in large quantities, the amount of end carboxyl groups increases, consequently spinning stability and weatherability tend to fall. As the melting point of desirable polymer, it is 230 degrees C or more, and is 233 degrees C or more still more preferably.

[0012] 0.4-1.5 are desirable still more desirable, and the limiting viscosity  $[\eta]$  of the polymer used for this invention is 0.7-1.2. In this range, fiber excellent in intensity and spinning nature can be obtained. The limiting viscosity  $[\eta]$  in this invention is a value calculated based on the following definition formula.

$$[\eta] = \lim_{C \rightarrow 0} 1 / (C \times (\eta_{\text{tar}} - 1))$$

$\eta_{\text{tar}}$  in a  $C \rightarrow 0$  definition formula is the value which broke the 35-degree C viscosity of the diluted solution of the sample which dissolved by o-chlorophenol of 98% or more of purity by viscosity of the above-mentioned solvent itself measured at the same temperature, and is defined as relative viscosity. Moreover, C is a solute weight value by the gram unit in the 100ml of the above-mentioned solutions.

[0013] When limiting viscosity is less than 0.4, since the molecular weight of polymer is too low, an on-the-strength manifestation becomes difficult. Conversely, since melt viscosity is too high and stable spinning is not made when limiting viscosity exceeds 1.5, it is not desirable. one sort of metal acetate [ if an example desirable as a process of the polymer used for this invention is given, will use a terephthalic acid or a dimethyl terephthalate as a raw material, and / this / trimethylene glycol ], such as a calcium acetate, a magnesium acetate, zinc acetate, a cobaltous acetate, and manganese acetate, or two sorts or more -- 0.03 – 0.1wt% -- in addition, screw hydroxypropyl terephthalate is obtained at 90 – 98% of rates of an ester interchange under an ordinary pressure or pressurization Thus, in order to make the purpose of this invention attain, it is desirable to use the acetate of metals other than transition metals. next, one sort of polycondensation catalysts, such as titanium tetrapod isopropanal PIKISHIDO, titanium tetrapod butoxide, and an antimony trioxide, or two sorts or more -- 0.03 – 0.15wt% -- desirable -- 0.03 – 0.1wt% -- it adds and is made to react under reduced pressure at 250–270 degrees C The arbitrary stages of a polymerization and putting in a stabilizer before a polycondensation reaction preferably have desirable white degree, amount of polytrimethylene terphthalate oligomer, and molecular weight of a resin constituent from a viewpoint which can control the 300 or less organic amount of resources in the amount of specification. As a stabilizer in this case, pentavalence or/, and trivalent phosphorus compounds and a hindered phenol system compound are desirable.

[0014] As pentavalence or/, and trivalent phosphorus compounds, trimethyl phosphate, triethyl phosphate, tributyl phosphate, triphenyl phosphate, trimethyl phosphite, triethyl phosphite, tributyl phosphite, triphenyl phosphite, etc. are mentioned, and trimethyl phosphite is desirable especially. A hindered phenol system compound is a phenol system derivative which has the substituent which has steric hindrance in the adjoining position of a phenol system hydroxyl group, and is a compound which has one or more ester combination in a molecule.

[0015] Specifically Pentaerythritol-tetrakis [3 (3, 5-G tert butyl-4-hydroxyphenyl) Propionate], 1, 1, 3-tris (2-methyl-4-hydroxy-5-tert-buthylphenyl) butane, 1, 3, 5-trimethyl -2, 4, 6-tris (3, 5-G tert-butyl-4-hydroxy benzyl) benzene, 3, 9-screw {2-[3-(3-tert-butyl-4-hydroxy-5-methylphenyl) propionyloxy]-1 and 1-dimethyl ethyl}-tetraoxaspiro [ 2, 4, 8, and 10-] [5, 5] undecane, 1, 3, 5-tris (4-tert-butyl-3-hydroxy-2, 6-dimethylbenzene) isophthalic acid, A triethyl glycol-screw [3 (3-tert-butyl-5-methyl-4-hydroxyphenyl) Propionate], 1, a 6-hexandiol-screw [3-(3, 5-G tert-butyl-4-hydroxyphenyl) propionate], 2 Two - Thio-diethylene-screw [3 (3 5-G

tert butyl-4-hydroxyphenyl) Propionate] octadecyl-3-(3, 5-G tert-butyl-4-hydroxyphenyl) propionate] can be illustrated. Pentaerythritol-tetrakis [3 (3, 5-G tert butyl-4-hydroxyphenyl) Propionate] is desirable especially.

[0016] It has the property generally shown below in the polymer obtained by the above-mentioned method. The content of the screw-3-hydroxypropyl ether by which copolymerization was carried out is less than [ 0.1wt% ]. Moreover, when b value shows a hue, depending on 10 or less and the case, it is three or less. Moreover, the amount of end carboxyl groups is 10-35mg Eq/kg. The fiber which constitutes the polyester super-thin fiber web of this invention is the range of the 0.8-5-micrometer diameter of average fiber, and is mixed fiber which has the moderate diameter distribution of fiber. While the intensity of the fiber from which the diameter of average fiber is obtained by less than 0.8 micrometers becomes inadequate, the fall of coloring nature and robustness takes place. On the other hand, if 5 micrometers is exceeded, it will become only the web which gives a scarce crude feel to flexibility. Moreover, since this super-thin fiber has the very small diameter of fiber, although it is difficult to measure the average length of fiber, in the case of 30mm or more and many, about 70-350mm can be presumed.

[0017] Generally the amount of eyes of the polyester super-thin fiber web of this invention is 5 - 200 g/m<sup>2</sup>, although it can set up arbitrarily by the use. It is a range. The polyester super-thin fiber web of this invention has the limiting viscosity of 0.4-1.0. For this reason, its intensity is high although the web obtained is rich in flexibility. Less than in 0.4, intensity is low and a polymer ball arises. On the other hand, the fiber which constitutes a web or more from 1.0 tangles, it results ill-behaved, and a pilling arises. It is the range of 0.6-0.8 from a viewpoint that intensity and front-face nature are excellent preferably [ both ].

[0018] The desirable manufacturing method of the polyester super-thin fiber web of this invention is explained below based on a drawing. After a suitable filter is therefore filtered, the polymer style by which melting was carried out within the extruder is led to the melting polymer induction (2) of a melt blow die (1), and is breathed out from an orifice-like nozzle (3) after that. The heating gas introduced into heating gas induction (4) simultaneously with it is led to the heating gas jet slit (5) formed of the melt blow die (1) and the lip (6), it blows off from here, this is \*\*ized in the aforementioned regurgitation polymer liquid, and super-thin fiber is formed. Subsequently, the uptake of this is carried out to the shape of a sheet, and a web is formed.

[0019] In this invention, it is important to make melting knockout temperature of

polymer into 260–300 degrees C. If 300 degrees C is exceeded, polymer will cause acceleration of heat deterioration and hydrolysis etc., melt viscosity will fall, the intensity of the web obtained will fall, and also the fall of generation of a polymer ball, eyes spots, and dyeing robustness is caused. Next, it is important to make into 270–320 degrees C temperature of the elevated-temperature high-speed gas style spouted in order to blow away and make a polymer style super-thin in this invention. At less than 270 degrees C, since the cooling effect over a regurgitation polymer style becomes excessive, \*\*izing [ of a polymer style ] will become inadequate, a polymer ball will increase, and the quality of a web will be reduced sharply. On the other hand, if 320 degrees C is exceeded, the temperature of melting polymer induction (2) and an orifice-like nozzle (3) will exceed 300 degrees C with the heat transfer from this gas. Preferably, it is 270–290 degrees C. As blow gas to be used, degradation of polymer has steam and advantageous air also from a cost side few.

[0020] The injection pressure of the elevated-temperature high-speed gas spouted in this invention is 2kg [ 0.3–4 //cm ]. It is desirable to set it as the range. In addition, an injection pressure is the value measured at the point near the lip (6) of heating gas induction (4). This injection pressure is 0.3 kg/cm<sup>2</sup>. Since the polymer \*\*ized energy of a jet gas becomes small in being the following, \*\*izing becomes poor. Therefore, the fall of flexibility and eyes spots happen. On the other hand, it is 4 kg/cm<sup>2</sup>. Above, a part of web by which coloring nature and robustness fell by \*\*ization of polymer progressing although the intensity of a web became strong and it became a desirable direction, and the uptake was carried out since the force of a jet gas was too strong will be able to wind up, and surface grace will be inferior. Such a phenomenon becomes easy to happen in polytrimethylene terphthalate, although not generated in a polyethylene terephthalate.

[0021]

[The operation gestalt of invention] Although an example etc. is given and this invention is hereafter explained more to a detail, needless to say, this invention is not limited at all by the example etc. In addition, the main measured value in an example was measured by the following methods.

(1) The limiting viscosity [eta] of \*\*\*\*\* is a value calculated based on the following definition formula.

$$[\eta] = \lim_{C \rightarrow 0} 1 / (Cx (\eta_{\text{tar}} - 1))$$

etar in a C->0 definition formula is the value which broke the 35-degree C viscosity of the diluted solution of the sample which dissolved by o-chlorophenol of 98% or more of purity by viscosity of the above-mentioned solvent itself measured at the

same temperature, and is defined as relative viscosity. Moreover, C is a solute weight value by the gram unit in the 100ml of the above-mentioned solutions.

[0022] (2) Using DSC by the melting point SEIKO electronic company, by the programming rate of 20 degrees C/min, it is nitrogen air current Shimonaka of 100 ml/min, and measured. Here, peak value of the peak of dissolution was made into the melting point.

(3) Chloroform extracted the polyester resin constituent and polyester fiber into which PTT oligomer turned minutely [ fixed quantity ] for 50 hours using the Soxhlet extractor, and weight % to the sample using the oligomer contained in the obtained residue showed them.

[0023] (4) It asked for a polyester resin constituent and the with a molecular weight of 300 or less contained in fiber organic substance from the chloroform liquid obtained in the structure determination and the fixed quantity (3) of the with a molecular weight of 300 or less organic substance. It analyzed using the gas chromatography equipped with the capillary column. The used column used two sorts, a silicon system and a polyethylene-glycol system. It asked for the concentration to the sample which the weighing capacity created the calibration curve to structure determination, and was used for it about each separated component using the mass spectrum (GC-MS) in ppm.

[0024] (5) About ten places with the arbitrary diameter sample of average fiber, ten-sheet photography was performed by one 2000 times the scale factor of this with the electron microscope. About one photograph, the diameter of ten arbitrary fiber was measured and ten photographs were followed in this. A total of 100 diameter measured value of fiber was calculated, and the average was calculated.

(6) The 10cmx10cm sample was continuously cut off over the width direction of an eyes spots web, and this weight was measured. The difference R of the average A of the value, maximum, and the minimum value was searched for, and eyes spots were measured by the following formula.

Eyes spots =  $R/A \times 100$ . [0025] (7) It evaluated using the degree cantilever method of adaptability (45 degrees). It is shown that flexibility is so high that a numeric value is small.

(8) In the cheesecloth, the 400g load was hung and the wear \*\*\*\*\* sheet was worn out 500 times. The front face was observed and surface change was observed.

(9) Hyperchromicity and \*\*\*\*\* KAYARON polyester blue 3RSF (Nippon Kayaku Co., Ltd. make : tradename) used owf 10%, dace spar tangent line(Akinari chemistry company make : tradename) 1g/l., a bath ratio 1:50, and pH were made the

dispersant 5, and dyeing was performed in the state of boil for 1 hour. Coloring nature evaluated the thickness of a color by the 1-5th class. In this case, it is shown that hyperchromicity is excellent, so that a number goes up. The dyeing object was rubbed 200 times, hanging a 200g load by the wet cheesecloth using a \*\*\*\* type friction tester, after drying a dyeing object, and the 1-5th class estimated the color projection to a cheesecloth. In this case, it is shown that there are so few color changes that a number goes up.

[0026] (Examples 1 and 2) The dimethyl terephthalate and 1 and 3-propanediol were taught by the mole ratio of 1:2, the mixture (9:1) of the calcium acetate equivalent to 0.1wt(s)% of the amount of theoretical polymer and a cobaltous acetate was added, the temperature up was carried out gradually, and the ester exchange reaction was completed at 240 degrees C. the obtained ester-interchange object -- titanium tetrapod butoxide -- 0.1wt(s)% of the amount of theoretical polymer -- it added and was made to react at 270 degrees C for 2 hours The limiting viscosity of the obtained polymer was 0.7. The amount of oligomer was 0.1wt(s)%, the with a molecular weight of 300 or less organic amount of resources was 330 ppm, and the melting point was 234 degrees C. Spinning did not have the thread breakage, either and did not have a fluff, either.

[0027] Dissolve the obtained polymer at 290 degrees C using an extruder, melting polymer is made to breathe out from the orifice of 0.3mmphi located in a line with the 1500-piece single tier in 1mm pitch, and it is continuously accumulated on the uptake side you made it located in 60cm under a die orifice and where it moves, and is eyes 200 g/m2. It rolled round as a random web so that it might become. The melt blow conditions of examples 1 and 2 and the acquired physical properties of a web were shown in Table 1.

[0028] Next, they are 30 kg/cm2 from the orifice of the 0.2mm diameter arranged in a straight line in 3mm pitch while putting this random web on the wire gauze and drawing in by degree of vacuum 50mmHg from the lower part. It spurts [ which is spouted continuously / high-speed ] out all over a sheet, it is put in a pressure, and, subsequently they are 10 kg/cm2. It processed similarly by the pressure. The physical properties of the obtained sheet were also shown in Table 1. the web and sheet which were obtained are excellent in flexibility and wear nature, and can be dyed a dark color by the ordinary pressure, and there are few polymer balls -- it excelled

[0029] (Examples 1 and 2 of comparison) It experimented by changing melt blow conditions into a Table 1 publication using the polymer manufactured in the example 1. The physical properties of the obtained web and a sheet were shown in Table 1. In



any case, since the diameter of average fiber separated from the range of this invention, it was crude.

(Examples 3 and 4 of comparison) The same experiment as an example 1 was conducted using the polyethylene terephthalate (example 3 of comparison), and the polybutylene terephthalate (example 4 of comparison). It experimented by changing melt blow conditions into a Table 1 publication. The physical properties of the obtained web and a sheet were shown in Table 1. In the example 3 of comparison, coloring nature is bad and it turns out in the example 4 of comparison that

\*\*\*\*\* is bad.

[0030]

[Table 1]

実施例	メルトブロー条件		ウェブ物性				シート物性					
	スチーム温度	スチーム圧力	平均繊維径	極限粘度	4 <sup>th</sup> リヤ玉の発生	目付量	見かけ密度	引張強度	柔軟度	磨耗性	発色性	湿摩擦堅牢性
	℃	kg/cm <sup>2</sup>	μm			%	kg/cm <sup>3</sup>	kg/cm	mm		級	級
1	288	1.8	4.0	0.63	ほとんどなし	4	0.25	2.1	45	良好	5	3-4
2	280	1.9	9.5	0.67	"	5	0.24	2.0	44	"	5	3-4
比較例												
1	280	6.0	0.7	0.48	多い	5	0.30	2.3	40	不良	4	2
2	280	0.2	10.2	0.60	ほとんどなし	8	0.35	1.7	31	不良	5	3-4
3	300	9.0	9.5	0.59	"	5	0.22	2.6	53	不良	3	4
4	280	1.9	9.2	0.63	少ない	8	0.25	1.8	42	普通	5	1-2

[0031] (Example 5 of comparison) The example 1 was repeated except having used titanium tetrapod butoxide 0.1wt% instead of the mixture of a calcium acetate and a cobaltous acetate as an ester-interchange catalyst. The amount of oligomer of the obtained polymer was 3.5wt(s)%, the with a molecular weight of 300 or less organic amount of resources was 1700 ppm, and the melting point was 233 degrees C. Although spinning was performed using this polymer, the white organic substance deposited in the lip side, and it was admitted in response to the influence that white powder was mixed in a sheet. When white powder was analyzed, it turns out that it is dimerized annular oligomer.

[0032]

[Effect of the Invention] According to this invention, by the melt blowing method, it can excel in flexibility and wear nature, and can be dyed a dark color by the ordinary pressure, and the polytrimethylene terphthalate super-thin fiber web by the melt blowing method with few polymer balls can be offered. This web is useful on objects for garments, such as an artificial leather base fabric, a filter, a tape, a disposable

diaper, a PAPPU agent base fabric, work clothes, a wiping cross, a keeping-warm cotton pad, a padding cloth, a sheet, etc.

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[Translation done.]

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TECHNICAL FIELD

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[The technical field to which invention belongs] this invention relates to the polytrimethylene terphthalate super-thin fiber web by the melt blowing method. Furthermore, in detail, it excels in flexibility and wear nature, and can be dyed a dark color by the ordinary pressure, and a polymer ball is related with the polytrimethylene terphthalate super-thin fiber web by the few melt blowing method.

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PRIOR ART

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[Description of the Prior Art] About the spinning method of the polymer by the melt blowing method Industrial - and - engineering chemistry The 48th volume, No. 8, 1956 It is indicated by 1342-1346 pages and the super-thin fiber web which used the polyethylene terephthalate in it is introduced. It or subsequent ones, a polyethylene-terephthalate homopolymer, a polyethylene-terephthalate copolymer, Many patent application about the super-thin fiber web by the melt blowing method using the polybutylene terephthalate is made (JP,53-65471,A, JP,63-53309,B, JP,3-8855,A, JP,4-2850,A).

[0003] The polytrimethylene terphthalate which is made to carry out the polycondensation of the trimethylene glycol (1, 3-propanediol) to the lower-alcohol ester of the terephthalic acid represented by a terephthalic acid or the dimethyl terephthalate on the other hand, and is obtained is the epoch-making polymer having the outstanding elastic-recovery nature, a low elastic modulus (soft feeling), a property similar to a polyamide called dyeable, and lightfastness, heat set nature, dimensional stability and a performance similar to a polyethylene terephthalate called a low water absorption. Taking advantage of the feature, it is applied to the BCF carpet, the brush, the tennis gut, etc. (JP,9-3724,A, JP,8-173244,A, JP,5-262862,A).

[0004] If the melt blow super-thin fiber web using polytrimethylene terphthalate is obtained, although a web with the new feature is expectable, the present condition is that such [ until now ] a proposal is not made. Of course, in application of the polyester web by melt blow, the feature, and the industrial usefulness or industrial manufacturing method of a certain thing are not shown for what is shown as one of the examples of polymer by which many TORITORI methylene terephthalate was enumerated as an example of polyester.

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EFFECT OF THE INVENTION

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[Effect of the Invention] According to this invention, by the melt blowing method, it can excel in flexibility and wear nature, and can be dyed a dark color by the ordinary pressure, and the polytrimethylene terphthalate super-thin fiber web by the melt blowing method with few polymer balls can be offered. This web is useful on objects for garments, such as an artificial leather base fabric, a filter, a tape, a disposable diaper, a PAPPU agent base fabric, work clothes, a wiping cross, a keeping-warm cotton pad, a padding cloth, a sheet, etc.

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TECHNICAL PROBLEM

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[Problem(s) to be Solved by the Invention] It may be able to be imagined that it can carry out a melt blow easily on the conditions near a polyethylene terephthalate fundamentally since the molecular structure of polytrimethylene terphthalate is the structure which introduced one methylene into the ethylene portion of a polyethylene terephthalate. However, polytrimethylene terphthalate has difference, like many oligomer and low-molecular-weight impurities are included in a polymerization object with quite low thermal stability with a very quick crystal speed as compared with a polyethylene terephthalate, and only a crude web is obtained even if it applies the manufacture conditions in the case of being a polyethylene terephthalate as it is. [0006] The technical problem of this invention is applying the outstanding performance which polytrimethylene terphthalate's has to a super-thin fiber web, pulling out a new performance, and establishing the industrial manufacturing method. Consequently, the polytrimethylene terphthalate super-thin fiber web was excellent in flexibility and wear nature, could be dyed the dark color by the ordinary pressure, and found out the bird clapper to the web with few polymer balls. Although the polyester super-thin fiber web by the melt blowing method for could dye it the dark color by the ordinary pressure, and moreover having excelled in robustness is very useful to simplification of a dyeing process etc., the present condition is that there is no such [ until now ] thing substance.

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MEANS

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[Means for Solving the Problem] That is, this invention is a super-thin fiber web which consists of polytrimethylene terphthalate substantially, and offers the polyester super-thin fiber web which serves as a 0.8–5-micrometer diameter of average fiber from the super-thin fiber group which has the limiting viscosity of 0.4–1.0. The polymer used for this invention is polytrimethylene terphthalate which carries out the polycondensation of a terephthalic acid and the 1 and 3-propanediol substantially, and is obtained. Even if it is a polytrimethylene terphthalate homopolymer that it is substantial in this invention, it is shown that you may be the polytrimethylene terphthalate copolymer shown below. namely, the range which does not spoil the purpose and effect of this invention -- glycol components, such as acid components, such as an isophthalic acid, a succinic acid, an adipic acid, 2, 6-naphthalene dicarboxylic acid, and 5-sulfoisophtharate tetrabutyl POSUHONIUMU salt, and 1, 4-butanediol, 1, 6-hexandiol, cyclohexane dimethanol, epsilon-caprolactone, 4-hydroxybenzoic acid, polyoxy ethylene glycol, a polytetramethylene glycol, etc. -- less than [ 10wt% ] -- copolymerization may be carried out

[0008] Moreover, to polytrimethylene terphthalate, the need may be accepted, and various kinds of additives, for example, a flatting, a thermostabilizer, a defoaming agent, a ready coloring material, a flame retarder, an antioxidant, an ultraviolet ray absorbent, an infrared-absorption agent, a crystalline-nucleus agent, a fluorescent brightener, etc. may be copolymerized or mixed. As for the polymer used for this invention, it is desirable that the content of the oligomer of trimethyleneterephthalate is less than [ 3wt% ], it becomes possible to avoid an on-the-strength fall by this, and also required spinning stability is industrially securable. in addition, the oligomer to which 2–4 trimethyleneterephthalate units were usually connected with trimethyleneterephthalate oligomer -- it is -- a line -- you may be a cyclic structure



even if it is structure

[0009] In exceeding 3wt(s)%, when carrying out spinning, oligomer deposits in the circumference of a nozzle, and it becomes easy to generate a polymer ball, for example. In order to perform spinning to stability for a long time, less than [ 1.5wt% ] is less than [ 1wt% ] desirable still more preferably. furthermore -- the point that the fluff of the obtained fiber decreases -- less than [ 0.5wt% ] -- less than [ 0.3wt% ] is desirable and still more preferably natural -- an ideal target is non-\*\*\* In addition, a polymer ball is the cob-like polymer generated in the edge and center section of the ball-like polymer which has a diameter with a diameter [ of web composition fiber ] of about about 10 to 500 times, or fiber. After observing this polymer ball using a microscope, or giving the means of a press, a calender, and confounding processing and others to remaining as it is or a web for a web and raising the fiber density, it is detectable by dyeing this. If a polymer ball exists mostly, the use of the super-thin fiber web obtained will be restricted greatly, and it will become impossible especially as a base fabric for artificial leather using it.

[0010] Furthermore, into the polymer used for this invention, it is desirable that the content of the with a molecular weight of 300 or less organic substance is less than [ 1wt% ], and it does not color within the limits of this, or the performance of excelling in lightfastness can be secured. The with a molecular weight of 300 or less said here organic substance is the organic substance by which copolymerization is not carried out to polymer. According to examination of this invention persons, as the with a molecular weight of 300 or less organic substance, allyl alcohol, an acrolein, 2-butanol, a hexanol, a heptanol, a glycidyl methyl ether, the oxy-propylmethyl ether, etc. existed, and it found out that the total amount of these compounds had big influence on a moldability, product durability, and weatherability. When the content of the with a molecular weight of 300 or less organic substance exceeds 1wt%, it will become or a polymer ball will become being easy to generate what it is easy to color with light. Preferably, the content of the with a molecular weight of 300 or less organic substance is 5000 ppm or less, and is 1000 ppm or less especially preferably. Of course, it is non-\*\*\* ideally.

[0011] As the melting point of the polymer used for this invention, it is desirable that it is 227 degrees C or more. With the melting point, it is defined in 220-250 degrees C as the peak value of the peak considered to be dissolution here. When two or more dissolution peaks exist (a shoulder peak is also included), let the peak of low temperature be the melting point. At less than 227 degrees C, a weatherproof fall becomes easy for the melting point to take place. For example, if polytrimethylene

terphthalate is manufactured at once and solid state polymerization of the polymer is carried out at about 200 degrees C, the content of oligomer can be reduced greatly. However, if solid state polymerization is performed, the melting point of raw material polymer falls greatly, and will be in the state where no less than 225 degrees C are not fulfilled. Into such polymer, copolymerization of the screw-3-hydroxypropyl ether which a trimethylene glycol dimerizes and generates is carried out in large quantities, the amount of end carboxyl groups increases, consequently spinning stability and weatherability tend to fall. As the melting point of desirable polymer, it is 230 degrees C or more, and is 233 degrees C or more still more preferably.

[0012] 0.4-1.5 are desirable still more desirable, and the limiting viscosity  $[\eta]$  of the polymer used for this invention is 0.7-1.2. In this range, fiber excellent in intensity and spinning nature can be obtained. The limiting viscosity  $[\eta]$  in this invention is a value calculated based on the following definition formula.

$$[\eta] = \lim_{C \rightarrow 0} 1 / (Cx (\eta_{rel} - 1))$$

$\eta_{rel}$  in a  $C \rightarrow 0$  definition formula is the value which broke the 35-degree C viscosity of the diluted solution of the sample which dissolved by o-chlorophenol of 98% or more of purity by viscosity of the above-mentioned solvent itself measured at the same temperature, and is defined as relative viscosity. Moreover, C is a solute weight value by the gram unit in the 100ml of the above-mentioned solutions.

[0013] When limiting viscosity is less than 0.4, since the molecular weight of polymer is too low, an on-the-strength manifestation becomes difficult. Conversely, since melt viscosity is too high and stable spinning is not made when limiting viscosity exceeds 1.5, it is not desirable. one sort of metal acetate [ if an example desirable as a process of the polymer used for this invention is given, will use a terephthalic acid or a dimethyl terephthalate as a raw material, and / this / trimethylene glycol ], such as a calcium acetate, a magnesium acetate, zinc acetate, a cobaltous acetate, and manganese acetate, or two sorts or more -- 0.03 - 0.1wt% -- in addition, screw hydroxypropyl terephthalate is obtained at 90 - 98% of rates of an ester interchange under an ordinary pressure or pressurization Thus, in order to make the purpose of this invention attain, it is desirable to use the acetate of metals other than transition metals. next, one sort of polycondensation catalysts, such as titanium tetrapod isopropanal PIKISHIDO, titanium tetrapod butoxide, and an antimony trioxide, or two sorts or more -- 0.03 - 0.15wt% -- desirable -- 0.03 - 0.1wt% -- it adds and is made to react under reduced pressure at 250-270 degrees C The arbitrary stages of a polymerization and putting in a stabilizer before a polycondensation reaction preferably have desirable white degree, amount of polytrimethylene terphthalate

oligomer, and molecular weight of a resin constituent from a viewpoint which can control the 300 or less organic amount of resources in the amount of specification. As a stabilizer in this case, pentavalence or/, and trivalent phosphorus compounds and a hindered phenol system compound are desirable.

[0014] As pentavalence or/, and trivalent phosphorus compounds, trimethyl phosphate, triethyl phosphate, tributyl phosphate, triphenyl phosphate, trimethyl phosphite, triethyl phosphite, tributyl phosphite, triphenyl phosphite, etc. are mentioned, and trimethyl phosphite is desirable especially. A hindered phenol system compound is a phenol system derivative which has the substituent which has steric hindrance in the adjoining position of a phenol system hydroxyl group, and is a compound which has one or more ester combination in a molecule.

[0015] Specifically Pentaerythritol-tetrakis [3 (3, 5-G tert butyl-4-hydroxyphenyl) Propionate], 1, 1, 3-tris (2-methyl-4-hydroxy-5-tert-buthylphenyl) butane, 1, 3, 5-trimethyl -2, 4, 6-tris (3, 5-G tert-butyl-4-hydroxy benzyl) benzene, 3, 9-screw {2-[3-(3-tert-butyl-4-hydroxy-5-methylphenyl) propionyloxy]-1 and 1-dimethyl ethyl}-tetraoxaspiro [2, 4, 8, and 10-] [5, 5] undecane, 1, 3, 5-tris (4-tert-butyl-3-hydroxy-2, 6-dimethylbenzene) isophthalic acid, A triethyl glycol-screw [3 (3-tert-butyl-5-methyl-4-hydroxyphenyl) Propionate], 1, a 6-hexandiol-screw [3-(3, 5-G tert-butyl-4-hydroxyphenyl) propionate], 2 Two - Thio-diethylene-screw [3 (3 5-G tert butyl-4-hydroxyphenyl) Propionate] octadecyl-3-(3, 5-G tert-butyl-4-hydroxyphenyl) propionate] can be illustrated. Pentaerythritol-tetrakis [3 (3, 5-G tert butyl-4-hydroxyphenyl) Propionate] is desirable especially.

[0016] It has the property generally shown below in the polymer obtained by the above-mentioned method. The content of the screw-3-hydroxypropyl ether by which copolymerization was carried out is less than [ 0.1wt% ]. Moreover, when b value shows a hue, depending on 10 or less and the case, it is three or less. Moreover, the amount of end carboxyl groups is 10-35mg Eq/kg. The fiber which constitutes the polyester super-thin fiber web of this invention is the range of the 0.8-5-micrometer diameter of average fiber, and is mixed fiber which has the moderate diameter distribution of fiber. While the intensity of the fiber from which the diameter of average fiber is obtained by less than 0.8 micrometers becomes inadequate, the fall of coloring nature and robustness takes place. On the other hand, if 5 micrometers is exceeded, it will become only the web which gives a scarce crude feel to flexibility. Moreover, since this super-thin fiber has the very small diameter of fiber, although it is difficult to measure the average length of fiber, in the case of 30mm or more and many, about 70-350mm can be presumed.

[0017] Generally the amount of eyes of the polyester super-thin fiber web of this invention is 5 – 200 g/m<sup>2</sup>, although it can set up arbitrarily by the use. It is a range. The polyester super-thin fiber web of this invention has the limiting viscosity of 0.4–1.0. For this reason, its intensity is high although the web obtained is rich in flexibility. Less than in 0.4, intensity is low and a polymer ball arises. On the other hand, the fiber which constitutes a web or more from 1.0 tangles, it results ill-behaved, and a pilling arises. It is the range of 0.6–0.8 from a viewpoint that intensity and front-face nature are excellent preferably [ both ].

[0018] The desirable manufacturing method of the polyester super-thin fiber web of this invention is explained below based on a drawing. After a suitable filter is therefore filtered, the polymer style by which melting was carried out within the extruder is led to the melting polymer induction (2) of a melt blow die (1), and is breathed out from an orifice-like nozzle (3) after that. The heating gas introduced into heating gas induction (4) simultaneously with it is led to the heating gas jet slit (5) formed of the melt blow die (1) and the lip (6), it blows off from here, this is \*\*ized in the aforementioned regurgitation polymer liquid, and super-thin fiber is formed. Subsequently, the uptake of this is carried out to the shape of a sheet, and a web is formed.

[0019] In this invention, it is important to make melting knockout temperature of polymer into 260–300 degrees C. If 300 degrees C is exceeded, polymer will cause acceleration of heat deterioration and hydrolysis etc., melt viscosity will fall, the intensity of the web obtained will fall, and also the fall of generation of a polymer ball, eyes spots, and dyeing robustness is caused. Next, it is important to make into 270–320 degrees C temperature of the elevated-temperature high-speed gas style spouted in order to blow away and make a polymer style super-thin in this invention. At less than 270 degrees C, since the cooling effect over a regurgitation polymer style becomes excessive, \*\*izing [ of a polymer style ] will become inadequate, a polymer ball will increase, and the quality of a web will be reduced sharply. On the other hand, if 320 degrees C is exceeded, the temperature of melting polymer induction (2) and an orifice-like nozzle (3) will exceed 300 degrees C with the heat transfer from this gas. Preferably, it is 270–290 degrees C. As blow gas to be used, degradation of polymer has steam and advantageous air also from a cost side few.

[0020] The injection pressure of the elevated-temperature high-speed gas spouted in this invention is 2kg [ 0.3–4 //cm ]. It is desirable to set it as the range. In addition, an injection pressure is the value measured at the point near the lip (6) of heating gas induction (4). This injection pressure is 0.3 kg/cm<sup>2</sup>. Since the polymer \*\*ized energy

of a jet gas becomes small in being the following, \*\*-izing becomes poor. Therefore, the fall of flexibility and eyes spots happen. On the other hand, it is 4 kg/cm<sup>2</sup>. Above, a part of web by which coloring nature and robustness fell by \*\*-ization of polymer progressing although the intensity of a web became strong and it became a desirable direction, and the uptake was carried out since the force of a jet gas was too strong will be able to wind up, and surface grace will be inferior. Such a phenomenon becomes easy to happen in polytrimethylene terphthalate, although not generated in a polyethylene terephthalate.

[0021]

[The operation gestalt of invention] Although an example etc. is given and this invention is hereafter explained more to a detail, needless to say, this invention is not limited at all by the example etc. In addition, the main measured value in an example was measured by the following methods.

(1) The limiting viscosity [ $\eta$ ] of \*\*\*\*\* is a value calculated based on the following definition formula.

$$[\eta] = \lim_{C \rightarrow 0} 1 / (C \times (\eta_{\text{tar}} - 1))$$

$\eta_{\text{tar}}$  in a  $C \rightarrow 0$  definition formula is the value which broke the 35-degree C viscosity of the diluted solution of the sample which dissolved by o-chlorophenol of 98% or more of purity by viscosity of the above-mentioned solvent itself measured at the same temperature, and is defined as relative viscosity. Moreover, C is a solute weight value by the gram unit in the 100ml of the above-mentioned solutions.

[0022] (2) Using DSC by the melting point SEIKO electronic company, by the programming rate of 20 degrees C/min, it is nitrogen air current Shimonaka of 100 ml/min, and measured. Here, peak value of the peak of dissolution was made into the melting point.

(3) Chloroform extracted the polyester resin constituent and polyester fiber into which PTT oligomer turned minutely [ fixed quantity ] for 50 hours using the Soxhlet extractor, and weight % to the sample using the oligomer contained in the obtained residue showed them.

[0023] (4) It asked for a polyester resin constituent and the with a molecular weight of 300 or less contained in fiber organic substance from the chloroform liquid obtained in the structure determination and the fixed quantity (3) of the with a molecular weight of 300 or less organic substance. It analyzed using the gas chromatography equipped with the capillary column. The used column used two sorts, a silicon system and a polyethylene-glycol system. It asked for the concentration to the sample which the weighing capacity created the calibration curve to structure determination, and

was used for it about each separated component using the mass spectrum (GC-MS) in ppm.

[0024] (5) About ten places with the arbitrary diameter sample of average fiber, ten-sheet photography was performed by one 2000 times the scale factor of this with the electron microscope. About one photograph, the diameter of ten arbitrary fiber was measured and ten photographs were followed in this. A total of 100 diameter measured value of fiber was calculated, and the average was calculated.

(6) The 10cmx10cm sample was continuously cut off over the width direction of an eyes spots web, and this weight was measured. The difference R of the average A of the value, maximum, and the minimum value was searched for, and eyes spots were measured by the following formula.

Eyes spots =  $R/A \times 100$ . [0025] (7) It evaluated using the degree cantilever method of adaptability (45 degrees). It is shown that flexibility is so high that a numeric value is small.

(8) In the cheesecloth, the 400g load was hung and the wear \*\*\*\*\* sheet was worn out 500 times. The front face was observed and surface change was observed.

(9) Hyperchromicity and \*\*\*\*\* KAYARON polyester blue 3RSF (Nippon Kayaku Co., Ltd. make : tradename) used owf 10%, dace spar tangent line(Akinari chemistry company make : tradename) 1g/l., a bath ratio 1:50, and pH were made the dispersant 5, and dyeing was performed in the state of boil for 1 hour. Coloring nature evaluated the thickness of a color by the 1-5th class. In this case, it is shown that hyperchromicity is excellent, so that a number goes up. The dyeing object was rubbed 200 times, hanging a 200g load by the wet cheesecloth using a \*\*\*\* type friction tester, after drying a dyeing object, and the 1-5th class estimated the color projection to a cheesecloth. In this case, it is shown that there are so few color changes that a number goes up.

[0026] (Examples 1 and 2) The dimethyl terephthalate and 1 and 3-propanediol were taught by the mole ratio of 1:2, the mixture (9:1) of the calcium acetate equivalent to 0.1wt(s)% of the amount of theoretical polymer and a cobaltous acetate was added, the temperature up was carried out gradually, and the ester exchange reaction was completed at 240 degrees C. the obtained ester-interchange object -- titanium tetrapod butoxide -- 0.1wt(s)% of the amount of theoretical polymer -- it added and was made to react at 270 degrees C for 2 hours The limiting viscosity of the obtained polymer was 0.7. The amount of oligomer was 0.1wt(s)%, the with a molecular weight of 300 or less organic amount of resources was 330 ppm, and the melting point was 234 degrees C. Spinning did not have the thread breakage, either and did not have a

fluff, either.

[0027] Dissolve the obtained polymer at 290 degrees C using an extruder, melting polymer is made to breathe out from the orifice of 0.3mmphi located in a line with the 1500-piece single tier in 1mm pitch, and it is continuously accumulated on the uptake side you made it located in 60cm under a die orifice and where it moves, and is eyes 200 g/m<sup>2</sup>. It rolled round as a random web so that it might become. The melt blow conditions of examples 1 and 2 and the acquired physical properties of a web were shown in Table 1.

[0028] Next, they are 30 kg/cm<sup>2</sup> from the orifice of the 0.2mm diameter arranged in a straight line in 3mm pitch while putting this random web on the wire gauze and drawing in by degree of vacuum 50mmHg from the lower part. It spurts [ which is spouted continuously / high-speed ] out all over a sheet, it is put in a pressure, and, subsequently they are 10 kg/cm<sup>2</sup>. It processed similarly by the pressure. The physical properties of the obtained sheet were also shown in Table 1. the web and sheet which were obtained are excellent in flexibility and wear nature, and can be dyed a dark color by the ordinary pressure, and there are few polymer balls -- it excelled

[0029] (Examples 1 and 2 of comparison) It experimented by changing melt blow conditions into a Table 1 publication using the polymer manufactured in the example 1. The physical properties of the obtained web and a sheet were shown in Table 1. In any case, since the diameter of average fiber separated from the range of this invention, it was crude.

(Examples 3 and 4 of comparison) The same experiment as an example 1 was conducted using the polyethylene terephthalate (example 3 of comparison), and the polybutylene terephthalate (example 4 of comparison). It experimented by changing melt blow conditions into a Table 1 publication. The physical properties of the obtained web and a sheet were shown in Table 1. In the example 3 of comparison, coloring nature is bad and it turns out in the example 4 of comparison that

\*\*\*\*\* is bad.

[0030]

[Table 1]

実施例	メルトブロー条件		ウェブ物性				シート物性					
	スチーム温度	スチーム圧力	平均繊維径	極限粘度	4°リヤ玉の発生	目付量	見かけ密度	引張強度	柔軟度	磨耗性	発色性	湿摩擦堅牢性
	℃	kg/cm <sup>2</sup>	μm			%	kg/cm <sup>3</sup>	kg/cm	mm		級	級
1	288	1.8	4.0	0.63	ほとんどなし	4	0.25	2.1	45	良好	5	3-4
2	280	1.3	9.5	0.67	"	5	0.24	2.0	44	"	5	3-4
比較例												
1	280	6.0	0.7	0.48	多い	5	0.30	2.3	40	不良	4	2
2	280	0.2	10.2	0.60	ほとんどなし	8	0.35	1.7	31	不良	5	3-4
3	300	9.0	3.5	0.59	"	5	0.22	2.6	53	不良	3	4
4	280	1.3	3.2	0.68	少ない	8	0.25	1.8	42	普通	5	1-2

[0031] (Example 5 of comparison) The example 1 was repeated except having used titanium tetrapod butoxide 0.1wt% instead of the mixture of a calcium acetate and a cobaltous acetate as an ester-interchange catalyst. The amount of oligomer of the obtained polymer was 3.5wt(s)%, the with a molecular weight of 300 or less organic amount of resources was 1700 ppm, and the melting point was 233 degrees C. Although spinning was performed using this polymer, the white organic substance deposited in the lip side, and it was admitted in response to the influence that white powder was mixed in a sheet. When white powder was analyzed, it turns out that it is dimerized annular oligomer.

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[Translation done.]



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DESCRIPTION OF DRAWINGS

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[Brief Description of the Drawings]

[Drawing 1] It is drawing on which the die circumference of the melt blow equipment explained by this invention was drawn roughly.

[Description of Notations]

- 1 Melt Blow Die
- 2 Melting Polymer Induction
- 3 Orifice-like Nozzle
- 4 Heating Gas Induction
- 5 Heating Gas Jet Slit
- 6 Lip

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[Translation done.]

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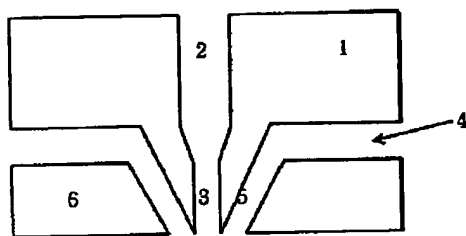
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DRAWINGS

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[Drawing 1]

図 1



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[Translation done.]

=> file caplus  
COST IN U.S. DOLLARS

SINCE FILE	TOTAL
ENTRY	SESSION
1.26	1.26

FULL ESTIMATED COST

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FILE COVERS 1907 - 25 Apr 2003 VOL 138 ISS 18  
FILE LAST UPDATED: 24 Apr 2003 (20030424/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s polyethylene terephthalate or polytrimethylene terephthalate

289793 POLYETHYLENE  
9635 POLYETHYLENES  
292619 POLYETHYLENE  
(POLYETHYLENE OR POLYETHYLENES)  
81076 TEREPHTHALATE  
1935 TEREPHTHALATES  
81422 TEREPHTHALATE  
(TEREPHTHALATE OR TEREPHTHALATES)  
22528 POLYETHYLENE TEREPHTHALATE  
(POLYETHYLENE (W) TEREPHTHALATE)  
660 POLYTRIMETHYLENE  
81076 TEREPHTHALATE  
1935 TEREPHTHALATES  
81422 TEREPHTHALATE  
(TEREPHTHALATE OR TEREPHTHALATES)  
545 POLYTRIMETHYLENE TEREPHTHALATE  
(POLYTRIMETHYLENE (W) TEREPHTHALATE)

L1 23030 POLYETHYLENE TEREPHTHALATE OR POLYTRIMETHYLENE TEREPHTHALATE

=> s fibers or fibres

462555 FIBERS  
1616 FIBRES  
L2 462935 FIBERS OR FIBRES

=> s l1 and l2

L3 5758 L1 AND L2

=> s web

21773 WEB  
5952 WEBS  
L4 24385 WEB  
(WEB OR WEBS)

=> s net or mesh or network

88054 NET

4932 NETS  
 90816 NET  
     (NET OR NETS)  
 73476 MESH  
     2736 MESHERS  
 75286 MESH  
     (MESH OR MESHERS)  
 107096 NETWORK  
     39594 NETWORKS  
 126228 NETWORK  
     (NETWORK OR NETWORKS)  
 L5 288751 NET OR MESH OR NETWORK

=> d his

(FILE 'HOME' ENTERED AT 14:00:07 ON 25 APR 2003)

FILE 'CAPLUS' ENTERED AT 14:03:37 ON 25 APR 2003

L1 23030 S POLYETHYLENE TEREPHTHALATE OR POLYTRIMETHYLENE TEREPHTHALATE  
 L2 462935 S FIBERS OR FIBRES  
 L3 5758 S L1 AND L2  
 L4 24385 S WEB  
 L5 288751 S NET OR MESH OR NETWORK

=> s 13 and 14 and 15

L6 7 L3 AND L4 AND L5

=> d 16 1-7 bib,abs

L6 ANSWER 1 OF 7 CAPLUS COPYRIGHT 2003 ACS

AN 2001:777997 CAPLUS

DN 135:332507

TI Nonwoven fabric wipers with good softness and wiping properties comprising poly(trimethylene terephthalate) **fibers**

IN Inagaki, Kenji; Yamazaki, Yasuyuki

PA Teijin Ltd., Japan

SO Jpn. Kokai Tokkyo Koho, 5 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 2001295169	A2	20011026	JP 2000-110505	20000412
PRAI	JP 2000-110505		20000412		

AB The nonwoven fabrics (A) are formed by water-spraying method and comprise .gtoreq.30% poly(trimethylene terephthalate) (I) staple **fibers** and have the forms fixed by means of having the surface of the nonwoven fabrics having bonded portion content 25-75% and/or having the nonwoven fabrics laminated with **net**-like sheets, and have basis wt. 15-200 g/m2, av. breaking strength in the warp or filling directions .gtoreq.8N/50 mm, and stiffness <10 cm, or the nonwoven fabrics comprise A nonwoven fabrics having av. denier per filament of I staple **fibers** .ltoreq.6.7 dtex. A carded **web** comprising 50:50 blend of I staple **fibers** with denier per filament 2 dtex and poly(ethylene terephthalate) **fibers** was sprayed with H2O at 490x104Pa and 980x104 Pa, dried, and embossed at 160.degree. to give a nonwoven fabric wiper having bonded portion content 50% (on nonwoven area) and having basis wt. 51 g/m2, av. strength 10.4 N/50 mm, and stiffness 5 cm and exhibiting good wiping properties.

L6 ANSWER 2 OF 7 CAPLUS COPYRIGHT 2003 ACS

AN 1999:803127 CAPLUS

DN 132:36943

TI Alkali-resistant nonwoven fabrics of polyester **fibers** coated with polyoxyethylenes containing alkylphenyl groups or polycyclic aromatic groups as lubricant finishes and suitable for construction materials

IN Adachi, Masataka

PA Toyobo Co., Ltd., Japan

SO Jpn. Kokai Tokkyo Koho, 4 pp.  
CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 11350325	A2	19991221	JP 1998-165218	19980612
PRAI	JP 1998-165218		19980612		
OS	MARPAT 132:36943				

AB The nonwoven fabrics comprise mech. interlaced polyester **fibers** and are finished with XO(CH<sub>2</sub>CH<sub>2</sub>O)nY [I; X = alkylphenyl, polycyclic arom. group; Y = H, SO<sub>3</sub>M (M = alkali metal, NH<sub>4</sub>); n .gtoreq.4] lubricants and have I content .gtoreq.0.1% (on fiber). Poly(ethylene terephthalate) was melt spun, piled on a **net**, hot-pressed to form a spunbonded **web**, sprayed with I (X = polycyclic arom. group; Y = H; n = 6) to corresponding I content 0.5%, kept 5 h at room temp., and dried to give a nonwoven fabric exhibiting wt. retention 82.4% after 48 h in 5 N NaOH at 80.degree..

L6 ANSWER 3 OF 7 CAPLUS COPYRIGHT 2003 ACS

AN 1999:698398 CAPLUS

DN 131:323817

TI Nonwoven fabrics from polyester conjugate **fibers** having styrene polymers, acrylate polymers or methylpentene polymers as the core and showing high stretchability and construction materials therefrom

IN Aoki, Masahiro; Ito, Tetsuya; Horiguchi, Yasuyoshi

PA Toray Industries, Inc., Japan

SO Jpn. Kokai Tokkyo Koho, 5 pp.  
CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 11302959	A2	19991102	JP 1998-113012	19980423
PRAI	JP 1998-113012		19980423		

AB The nonwovens comprise polyester conjugate **fibers** (A) contg. 0.1-15% component comprising styrene polymers, acrylate polymers, acrylate-styrene copolymers, and/or methylpentene polymers and exhibiting elongation .gtoreq.80% on stretching the **fibers** (length 200 mm) at 200 m/min or comprise A **fibers** and polyester **fibers**, polyamide **fibers**, polyolefin **fibers**, acrylic **fibers**, polyurethane **fibers**, and/or poly(Ph sulfone)-type **fibers** and are optionally reinforced with fabrics. Poly(ethylene terephthalate) as the sheath and polystyrene (Styron 685) as the core were together melt spun at 95:5 wt. ratio, passed through an ejector at 6000 m/min, piled on a **net**, and calendered at roll temp. 150.degree. to give a nonwoven **web** comprising **fibers** with elongation 130%. The nonwoven **web** was needlepunched to give a nonwoven fabric exhibiting elongation 100 and 120%, resp., in the machine and transverse directions and suitable for covering foundations with raised and depressed portions.

L6 ANSWER 4 OF 7 CAPLUS COPYRIGHT 2003 ACS

AN 1998:406258 CAPLUS

DN 129:57798

TI Wide-mesh textile **nets** for reinforcing bitumen-bonded coatings, especially road surfaces

IN Kassner, Juergen; Pintz, Heiko; Von Fransecky, Ulrich  
PA Huesker Synthetic G.m.b.H. und Co., Germany  
SO Ger. Offen., 6 pp.  
CODEN: GWXXBX

DT Patent  
LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 19652584	A1	19980618	DE 1996-19652584	19961217
	RU 2166019	C2	20010427	RU 1999-115877	19971205
	US 6503853	B1	20030107	US 1999-331282	19991004
	US 2003017771	A1	20030123	US 2002-245536	20020917
PRAI	DE 1996-19652584	A	19961217		
	WO 1997-EP6809	W	19971205		

AB The screens, coated with a bitumen-compatible adhesion-promoting material and consisting essentially of 2 groups of load-bearing strings of glass **fibers**, polymeric **fibers**, or polycondensate **fibers**, one in the machine direction and the other in the cross direction, the **nets** are machine-bonded to a thin **web**, and the binding yarn surround the strings running in the machine direction and immobilize the strings running in the cross direction. The strings may consist of bitumen-compatible, i.e., bitumen-bondable, **fibers**

RE.CNT 17 THERE ARE 17 CITED REFERENCES AVAILABLE FOR THIS RECORD  
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L6 ANSWER 5 OF 7 CAPLUS COPYRIGHT 2003 ACS

AN 1992:450741 CAPLUS

DN 117:50741

TI Applications of electrically conductive nonwoven textiles, and plastic articles suitable for use in electrostatic coating processes

PA Lantor B. V. te Veenendaal, Neth.

SO Neth. Appl., 8 pp.

CODEN: NAXXAN

DT Patent

LA Dutch

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	NL 9002073	A	19920416	NL 1990-2073	19900921
PRAI	NL 1990-2073		19900921		

AB The elec. conductive nonwoven textiles, at or in the surface of plastic articles, provide the cond. necessary in electrostatic coating processes. At least one of the surfaces of the plastic articles has been provided with an elec. conductive nonwoven **web**. The **fibers** of the **web** may have been metalized, or the **web** may have been provided with a **network** of metallic **fibers**. A needled nonwoven **web** of PBTP-PET (polyester) **fibers** (basis wt. 100 g/m<sup>2</sup>), prepressed at 4 g/cm<sup>2</sup> was laminated with a thermoprint-bonded **web** (basis wt. .apprx.35 g/cm<sup>2</sup>) consisting of 80% Ni-coated **fibers** and 20% PBTP-PETP **fibers**, using an interlayer of polyester threads (basis wt. .apprx.10 g/m<sup>2</sup>) provided every 5 cm with diagonally arranged Cu wire (diam. 0.1 mm). The resulting **web** had d. 0.48 g/cm<sup>3</sup> (measured under a pressure of 40 g/cm<sup>2</sup>, and surface resistivity <0.1 .OMEGA. measured over a length of 10 m. The **web** was pressed as surface layer with unsatd. polyester sheet-molding compds. to give a very smooth, low-porosity product that was electrostatically coated with polyester powder, and hardened at 180.degree. to obtain a homogeneous, smooth surface.

L6 ANSWER 6 OF 7 CAPLUS COPYRIGHT 2003 ACS

AN 1988:136532 CAPLUS

DN 108:136532

TI Process for manufacturing thin-walled structural ceramic articles  
 IN Disselbeck, Dieter; Stahl, Dieter  
 PA Hoechst A.-G., Fed. Rep. Ger.  
 SO Ger. Offen., 5 pp.  
 CODEN: GWXXBX  
 DT Patent  
 LA German  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 3622573	A1	19880107	DE 1986-3622573	19860704
	DE 3622573	C2	19960711		
PRAI	DE 1986-3622573		19860704		

AB Flat, large-surface textile structures made of fibrous materials are shaped into self-supporting articles with **net**-like, porous walls in the manuf. of the title articles by coating with a suspension contg. a finely divided sinterable powder, a volatile liq. phase, and a binder, followed by drying of the suspension, and heating until the powder is sintered and the **fibers** destroyed. The textile material is made of org. **fibers** and is provided in **mesh** form or as a honeycomb structure by bonding layers of the fibrous materials together and pulling them apart. The binder is a hardenable phenolic resin. A **polyethylene terephthalate** fiber-based **web** was impregnated with an aq. PhOH-HCHO resin soln., precondensed to a tack-free prepreg, and heated 1 min under pressure at 180.degree. to soften the binder and shape the prepreg into a profile plate having a regular **network** with fiber spacing of 0.5-0.8 mm. The plate was shaped into a cylinder having length 10 cm and diam. 3-4 cm, coated with a porcelain slip, predried 2 h at 110.degree., and fired at 1450.degree. without glazing to give a thin-walled porcelain cylinder in which the original **net**-like structure was barely discernible.

L6 ANSWER 7 OF 7 CAPLUS COPYRIGHT 2003 ACS  
 AN 1970:67702 CAPLUS  
 DN 72:67702  
 TI Low-density **web**-like cushioning structure of cellular filamentary material  
 IN Fish, Floyd H., Jr.; Stine, Cawley R.  
 PA du Pont de Nemours, E. I., and Co.  
 SO U.S., 12 pp.  
 CODEN: USXXAM  
 DT Patent  
 LA English  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 3485711	A	19691223	US 1966-559981	19660623
	BE 699822	A	19671116	BE 1967-699822	19670613
	NL 6708299	A	19671227	NL 1967-8299	19670615
	CH 678937	A4	19710514	CH 1967-893767	19670623
	CH 515369	A	19711115	CH 1967-515369	19670623
PRAI	US 1966-559981		19660623		

AB A resilient, low-d. cushioning structure, suitable as a carpet underlay, was prepd. from closed-cell, pneumatic, polymeric, cellular filamentary material. The material had superior cushioning properties at an exceptionally low wt./unit area. The compns. had an av. dynamic modulus (calcd. from the formula  $Z = 100 (.DELTA.L)/(C2 - C1)$ , where  $.DELTA.L = 5$  psi,  $C2 = \%$  compression at 25 psi, and  $C1 = \%$  compression at 20 psi) of 390 psi compared with 631, 583, 420, and 1288 psi for hair/jute, patterned sponge rubber, flat sponge rubber, and polyurethane, resp. Ultramicrocellular filaments were prepd. by extrusion of a foamable compn. contg. 400 g poly(ethylene terephthalate) (I) (relative viscosity 41, 8.7% in 70 parts 2,4,6-trichlorophenol and 100 parts PhOH), 350 ml CH<sub>2</sub>Cl<sub>2</sub>, 50 ml 1,1,2-trichloro-1,2,2-trifluoroethane, and 52 g perfluorocyclobutane.

The compn. was heated at 213.degree., connected to a 1200-psig N ballast tank, and extruded through a die with 0.003-in.-diam. holes. A sandwich of screens upstream from the extrusion holes were of 50-, 325 -, 325-, and 50-mesh, resp. The extruded filaments expanded to their max. diam. near the face of the die, then collapsed to a d. of 0.065 and were reexpanded by heating to their max. diam. of 0.010 in. The filaments were sprayed with water and randomly collected on an Al plate in a 3.5-in. batt. The batt was sliced to form squares, each .apprx.1/2 the original thickness. Expanded metal sheets were placed on 1 face of each sample, each metal sheet being .apprx.0.04 in. thick and having diamond-shaped openings, and each opening being sepd. from the next by a strip of metal .apprx.0.2 in. wide. The sample was embossed at room temp. in a hydraulic press, sprayed with a dil. soln. of a polyurethane adhesive in trichloroethylene, and expanded 1 hr at 120.degree.. The samples were thick, firm, and resilient. Batts of I were also coated with a latex of Foamtol BGL-9002 latex (a 58% dispersion in H2O of natural rubber and a butadiene-styrene copolymer) 365, FV-2003 (a 53% aq. dispersion of S and an accelerator) 20, FZ-3005 antioxidant (a 51% aq. dispersion of ZnO) 15 parts, Aquarex ME surfactant 100, Daxad 11 10, and distd. H2O 890 parts. A silencer soln. of 40% aq. poly(dimethylsiloxane) and poly(methylsiloxane) 9, Dow-Corning Catalyst 21 (a mixt. of Bu2Sn dilaurate and Zn 2-ethyl hexoate) 1, and distd. H2O was then applied. The cushioning products were also prepd. by drawing a heated blade across the surface of the batt to melt some of the polymer.

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L1	23030 S POLYETHYLENE TEREPHTHALATE OR POLYTRIMETHYLENE TEREPHTHALATE
L2	462935 S FIBERS OR FIBRES
L3	5758 S L1 AND L2
L4	24385 S WEB
L5	288751 S NET OR MESH OR NETWORK
L6	7 S L3 AND L4 AND L5

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